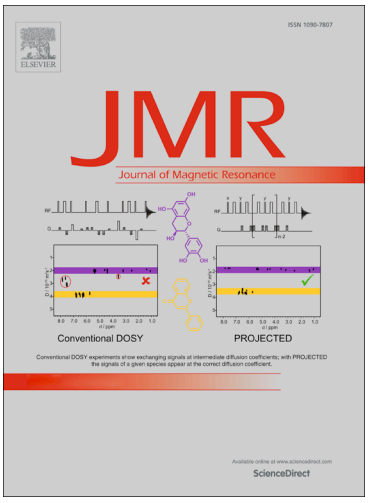

Setting the magic angle for fast magic-angle spinning probes

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Setting the magic angle for fast magic-angle spinning probes

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Abstract

Fast magic-angle spinning, coupled with ^{1}H detection is a powerful method to improve spectral resolution and signal to noise in solid-state NMR spectra. Commercial probes now provide spinning frequencies in excess of 100 kHz. Then, one has sufficient resolution in the ^{1}H dimension to directly detect protons, which have a gyromagnetic ratio approximately four times larger than ^{13}C spins. However, the gains in sensitivity can quickly be lost if the rotation angle is not set precisely. The most common method of magic-angle calibration is to optimize the number of rotary echoes, or sideband intensity, observed on a sample of KBr. However, this typically uses relatively low spinning frequencies, where the spinning of fast-MAS probes is often unstable, and detection on the ^{13}C channel, for which fast-MAS probes are typically not optimized. Therefore, we compare the KBr-based optimization of the magic angle with two alternative approaches: optimization of the splitting observed in ^{13}C-labeled glycine-ethylester on the carbonyl due to the Cα–C' *J*-coupling, or optimization of the H–N *J*-coupling spin echo in the protein sample itself. The latter method has the particular advantage that no separate sample is necessary for the magic-angle optimization.